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(54) Method and apparatus for recovering acetic acid from aqueous streams

Verfahren und Vorrichtung zur Wiedergewinnung der Essigsäure aus wässrigen Strömen

Procédé et appareillage pour récupération d'acide acétique à partir de courants aqueux

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Description

[0001] This invention relates to a method and apparatus for recovering acetic acid, which is the solvent typically used in the production of terephthalic acid and is also a recoverable waste stream in other important industrial processes. The method and apparatus provide a significant reduction of energy consumption, allow an increase in capacity for existing plants, and eliminate the organics emission problem currently existing in most terephthalic acid production plants.

[0002] Acetic acid is a recoverable solvent utilized in the production of terephthalic acid in many processes, see U.S. Patent 4,769,487, British 1,583,755, Canadian 1,113,957, and Japanese 53-71034, 58-39812, 59-33579, 53-79836, and 56-4587.

[0003] Distillation has been widely used as a primary operation for acetic acid recovery in such processes, utilizing one or more towers to process a number of streams of varying concentrations of acetic acid with the purpose of recovering it for further use in the oxidation step. The products from the distillation tower are a bottom stream of concentrated acetic acid, and an overhead stream that ideally would be pure water.

[0004] Because of the high non-ideality of the system acetic acid/water and the equilibrium limitation in such a system, it is necessary to utilize a distillation tower with a high number of theoretical stages and high reflux ratio to be able to obtain reasonably low levels of acetic acid in the distilled water.

[0005] These involve a high investment cost because of the large dimensions of the required equipment and a high operating cost because of the high steam consumption involved. Furthermore, the traditional process scheme does not allow one to obtain economically a distillate completely free of acetic acid. This limitation, in turn, presents two main problems: a cost associated with the operation resulting from the acetic acid losses, and an environmental problem that is continually increasing, because of the ever more rigorous standards for acceptable levels of emission to the environment.

[0006] There has been an effort to look for alternatives to solve the problem. Resort has been had to azeotropic distillation, involving the addition of an additional component to the distillation tower to improve the relative volatility of the separation and reduce the separation requirements. This existing option provides some reduction in the operating costs, but it generates some additional operating and environmental problems.

[0007] A study of the vapour liquid equilibrium of the components acetic acid/water shows the difficulty in the dilute acetic region of reducing the acetic acid in the distilled water. The achievement of a reduction from the typical design value of 0.5 wt% acetic acid overhead to 0.1 wt% acetic acid overhead requires an increase in reflux ratio of about 10-15% or the inclusion of several additional theoretical stages to maintain the same reflux ratio.

[0008] For several years it has been known that the use of liquid-liquid extraction is a way to recover acetic acid from dilute streams. Several extractive agents have been identified, and it is possible to economically recover acetic acid from streams containing 0.1 wt% acetic acid to 20% acetic acid. Some of the agents usually used are acetates, amines, ketones and phosphine oxides and mixtures thereof.

[0009] Once the extraction step is completed, a series of distillation steps are required to recover the acid and to recirculate the extractive agent back to the extraction stage. The level of impurities in the feed and the affinity of the particular extractive agent for water dictate the additional steps required in the system.

[0010] Japanese Patent Specification 63-156744 describes a system for recovering acetic acid contained in water by distilling the mixture to give an essentially acetic acid bottoms product and an essentially water top product which is then subjected to extraction with methyl acetate to provide separate water and acetic acid streams. However, the maximum acetic acid content in the essentially water top product is only 1.4%.

[0011] According to the invention in one aspect there is provided a method for separating acetic acid and water, the method being for use in a plant using a water solution of acetic acid, comprising:

(a) feeding at least one input acetic acid-containing water stream from the plant to a dehydration device and applying heat to the input stream received by the device to separate acetic acid from water in the device to produce thereby an output bottom stream of relatively concentrated acetic acid in water, and an output overhead stream of relatively dilute acetic acid in water,

(b) condensing acetic acid and water from the said output overhead stream to form an output overhead condensate, and

(c) feeding the output overhead condensate to a liquid-liquid extraction system and contacting it with a liquid extractant in a contactor to extract acetic acid from the condensate and thereby form a first contactor output stream containing acetic acid and extractant, and a second contactor output stream containing water, feeding the first contactor output stream to an extraction system separator device, and separating the acetic acid and the extractant therein to produce an extractant output stream for recycle to the contactor and an acetic acid output stream, characterised in that the said output overhead stream from the dehydration device has an acetic acid concentration of 2 to 20% by weight.

[0012] The present invention is based on the specific application of known unit operations technologies and use of extractive agents for acetic acid that independently of each other do not produce the major advan-

tages seen in the invention, but when these approaches are exploited together in the present arrangement, they provide a new process scheme with the benefits previously discussed.

[0013] A significant reduction of energy required (as measured by reflux ratio) can be obtained by relaxing the specification for the acetic acid overhead composition in the acetic acid recovery, still from 0.1 wt% acetic acid to 0.5 wt% acetic acid which can result in a 10-15% reduction in energy consumption. A further relaxation of the overhead acetic acid composition requirement from 0.5 wt% acetic acid to 10% acetic acid can result in a 40-50% reduction in energy consumption.

[0014] In accordance with the invention, a higher concentration of acetic acid in the overhead stream is accepted. Thus the reflux ratio is reduced, and the internal loadings of a typical acetic acid dehydration tower are reduced, representing a considerable increase in capacity.

[0015] The extraction system is capable of handling from 2 to 20% by weight acetic acid in the overhead stream and may use any of the known extraction solvents (ethyl acetate, other acetates, primary amines, secondary amines, ternary amines, MEK MiBK, other ketones, phosphine oxides). The preferred solvents are the phosphine oxides commercialized by Cytek (formerly American Cyanamid) under the Cyanex name, and amines.

[0016] According to the invention in another aspect there is provided an acetic acid and water separation system for use in a plant using a water solution of acetic acid, comprising:

(a) a dehydration device equipped to receive at least one input acetic acid-containing water stream from the plant and to apply heat to the input stream received by the device to separate water from acetic acid in the device thereby to produce an output bottom stream of relatively concentrated acetic acid in water, and an output overhead stream of acetic acid in water having an acetic acid concentration of 2 to 20% by weight,

(b) condensing means to liquify acetic acid and water from the output overhead stream to form an output overhead condensate, and

(c) a liquid-liquid extraction system comprising:

(i) a contactor to receive the output overhead condensate and contact it with a liquid extractant to extract acetic acid from the condensate and thereby form a first contactor output stream containing acetic acid and extractant, and a second contactor output stream containing water, and

(ii) an extraction system separator device equipped to receive the said first contactor output stream and separate the acetic acid and the extractant therein to produce an extractant output stream for recycle to the contactor and an acetic acid output stream,

characterised in that means are provided for feeding at least one additional relatively dilute acetic acid stream from the plant to the liquid-liquid extraction system along with the output overhead condensate from the dehydration device.

[0017] Since the operating requirements of the acetic acid dehydration column have been changed drastically from the requirements in the prior art, it is also possible to feed diluted acid streams (0.5% wt% - 40 wt%) previously sent to the dehydration column directly to the extraction system. This represents a large energy savings for the overall solvent recovery system, because the water in these streams need not be evaporated, and it will give an additional capacity increase for the dehydration column.

[0018] To increase capacity in a limited dehydration column, a new column is typically needed. For a 300,000 MTY plant, the investment in a new column for a 30% increase capacity can be between 6-8 million dollars. The system of the invention may be built for about half of that amount and achieve not only the capacity increase, but also reduction of emissions to the atmosphere.

[0019] In a preferred embodiment of the present invention scheme high pressure and low pressure absorption systems can be included in the recovery system. In current designs of typical terephthalic acid production plants, N_2 is introduced to the dehydration system. This creates a stream of non-condensables that entrains acetic acid, and some acetic acid is eventually carried to the atmosphere. In addition, the typical design presents a vent in the overhead vapour line that continuously sends organics to the atmosphere.

[0020] In the invention, two ways to improve and reduce these emissions of organics are provided. A preferred arrangement is to use the liberated condensing capacity in the dehydration column to condense more of the organics in conjunction with a new low pressure absorber system to remove the acids that are still not condensed. The new low pressure absorber system can handle all the low pressure vents from the plant and primarily the vent from the overhead vapour line and the overhead condenser vent line. The absorption liquid may be chilled water, diluted acetic acid (from a stream previously sent to dehydration) or preferably, a phosphine oxide solvent (Cyanex). In the case of using chilled water or diluted acetic acid, the bottom stream from the low pressure absorber may be sent to the new extraction unit. In the case of using the phosphine oxide, the stream is sent to the recovery tower of the extraction system.

[0021] Currently in a typical production plant design there are two high pressure absorbers used to recover organics from the oxidizer overheads. The first uses acetic acid primarily to recover p-xylene and methyl

acetate; and the second uses water from the overheads of the dehydration column to recover the remaining acetic acid. In a preferred embodiment of the invention both high pressure absorbers may be combined into one, if desired, or the second absorber may be changed to use phosphine oxide as the absorption solvent. This change eliminates a water recirculation stream in the plant from the high pressure absorber to the dehydration tower and back to the high pressure absorber. The benefits are an improvement in energy efficiency and capacity in the recovery system. The phosphine oxide stream from the one absorber or the secondary absorber reduces the emissions from the oxidizer overheads by improving absorption.

[0022] The combination of the high pressure absorber modification and the new low pressure absorber system helps terephthalic acid production plants to economically reduce atmospheric emission levels to values lower than those required by environmental agencies.

[0023] Another area where the invention presents great benefits for the typical production plant is in the water handling. At present the water stream most typically discharged to neutralization is the overhead of the dehydration column with acetic acid values between 0.2 wt% to 1 wt%. This represents a heavy and objectionable load on the neutralization/biological treatment section of the plant. The arrangement of the invention provides a significant reduction of acetic acid in the water levels to about 100 ppm to 500 ppm by weight, dependent on the plant requirements. (The expected solvent content is between 10 ppm wt. to 200 ppm wt.) Such water with this concentration can be used as cooling water or process water in the plant after a suitable treatment with activated carbon. Another major use of this water, for the case where the acetic acid content is low, can be as a solvent for the crystallization of terephthalic acid. This provides a considerable reduction of water usage in the plant by using the water produced in the oxidation step of terephthalic acid production for most of the plant needs.

[0024] In general, by including all aspects of the invention in the process by specific uses of known technologies, a greatly improved process for recovering the solvent in the production of terephthalic acid is achieved. The new process generates benefits of increased capacity, reduction of energy consumption and a great reduction in organic emissions to the atmosphere as well as to the treatment plant.

[0025] The dehydration device is preferably and most usually a column.

[0026] When the acetic acid and water separation system employs a liquid extractant which boils at a temperature higher than acetic acid, the acetic acid output stream from the extraction system separator column is an overhead stream therefrom, while on the other hand, when a liquid extractant which boils at a temperature lower than acetic acid is used, the acetic acid output stream from the extraction system separator column is a bottoms stream therefrom.

[0027] The said at least one additional relatively dilute acetic acid stream from the plant which is passed to the liquid-liquid extraction system along with the output overhead condensate stream from the dehydration column may be a bottoms stream from a high pressure absorber in the plant, or a condensate stream from a drying system.

[0028] The acetic acid and water separator system may also include a low pressure absorption unit adapted to contact an absorption solvent with at least one acetic acid containing vapour stream from the acetic acid and water separation system, and further adapted to feed absorption solvent after contact with the vapour stream to the liquid-liquid extraction system. The acetic acid containing vapour stream may be at least a portion of the output overhead stream from the dehydration column, or a portion of a vent stream from an output overhead condenser adapted to process the output overhead stream from the dehydration column, or may be an acetic acid containing vapour stream from a point in the plant other than the acetic acid and water separation system.

[0029] The acetic acid and water separator system may further comprise a high pressure absorber system equipped to contact an absorbent liquid at high pressure with a plant overhead vapour stream having acetic acid therein, and means for delivering the absorbent liquid, after contact with the plant overhead vapour stream to the separator system for processing therein. Those delivering means may be connected to deliver the absorbent liquid to the dehydration column or may be connected to deliver the absorbent liquid to the contactor of the liquid-liquid extraction system. Alternately, the delivering means may be connected to deliver the absorbent liquid to the extraction system separator column of the liquid-liquid extraction system.

[0030] The invention will now be described with reference to the accompanying drawings, in which:

FIG. 1 is a flow diagram of an acetic acid recovery system as typically found in the prior art;

FIG. 2A is a flow diagram for an extraction system using an extractive solvent boiling at relatively high temperature;

FIG. 2B is a flow diagram for an extraction system utilizing an extractive solvent which is relatively low temperature in boiling point;

FIG. 3 is a flow diagram of an acetic acid recovery system constructed in accordance with the invention, in which an extraction system is equipped to receive and process condensate from the overhead stream of the dehydration column of the system;

FIG. 4 is a flow diagram of an embodiment of the invention similar to that of FIG. 3, but in which relatively dilute acetic acid streams from plant locations are fed directly to the extraction system, rather than being fed to the dehydration column;

FIG. 5 is a flow diagram of a low pressure absorption unit which may be employed in accordance with certain embodiments of the invention;

FIG. 6 is a flow diagram of an acetic acid recovery system in accordance with the invention in which the low pressure absorber system of FIG. 5 is employed in connection with the basic system as illustrated in FIG. 3, for example;

FIG. 7 is a flow diagram of an acetic acid recovery system constructed in accordance with the invention in which the low pressure absorber system of FIG. 5 is employed in a form of the invention as depicted in FIG. 4;

FIG. 8 is a flow diagram of an acetic acid recovery system constructed in accordance with the invention and further including a high pressure absorption system whose bottom output stream is fed to the dehydration column of the system;

FIG. 9 is a flow diagram of an acetic acid recovery system constructed in accordance with the invention in which the bottom stream from the high pressure absorber system is fed to the extraction system that is provided to process condensate from the overhead stream of the dehydration column; and

FIG. 10 is a flow diagram of an acetic acid recovery system constructed in accordance with the invention in which the bottom stream from the high pressure absorber system is fed to the extraction system as in FIG. 9, but in which an extractant solvent is used instead of a stream from the dehydration column overhead.

[0031] FIG. 1 illustrates a flow diagram of a typical prior art acetic acid recovery system. The primary unit in the recovery system is a dehydration column 20. The column 20 receives input from several sources, one being the overhead from a stripper column 22, which in turn is fed mother liquor from the plant. The bottom stream from stripper column 22 is rich in acetic acid and is returned to the plant for reuse. The stripper column 22 has an optional condenser system 24.

[0032] Other feed input streams to the dehydration column 20 include line 26, which delivers bottom liquid from a high pressure absorber in the plant, line 28, which delivers liquid from a crystallizer, and line 30, which delivers water draw off liquid from the overhead stream out of an oxidation unit, such as is found in a typical terephthalic acid synthesis plant. Line 32 also delivers to the dehydration column 20 condensate from a drying system within the plant. The dehydration column 20 may be packed with structured or random packing or equipped with trays of various styles. Heat is supplied to the dehydration column 20 through reboiler 34 and the bottom stream out of the column is rich in acetic acid which is returned to the plant for reuse through line 36.

[0033] The overhead line 38 from the dehydration column 20 leads to a condenser 40 and a separation drum 42. The overhead line 38 may be equipped with a vent 44. The separation drum 42 may include a vent 46. The bottom stream from drum 42 is split into a reflux line 48, and an overhead product line 50, which is primarily water for delivery to the waste water treatment system. Line 50 may have a branch carrying a portion, or even all, of the water stream, which is not returned as reflux through line 48, to a high pressure absorber for further recovery of acetic acid. The line to the high pressure absorber is line 52.

[0034] Attention is now directed to FIGS. 2A and 2B, which illustrate extraction systems which may be employed in accordance with the invention. The extraction system of FIG. 2A is designed and adapted for use with an extraction solvent which is a "heavy boiler," that is, a solvent which boils at a temperature higher than the boiling point of acetic acid. This extraction system is designated generally 54A. The extraction system of FIG. 2B is one designed and equipped to utilize an extraction solvent which is a "light boiler," that is, one which boils at a lower temperature than acetic acid. In FIG. 2A, the extractor is designated 56, and receives dilute acetic acid through line 58 near its top. A bottom line 60 carries waste water to the waste water treatment plant or back into the terephthalic acid plant for reuse there. The overhead stream from the liquid-liquid contactor 56 is carried through line 62 to separator tower 64. The top stream out of separator tower 64 is carried by line 66 through condenser 68 and line 70 back to the plant for reuse. This stream is relatively highly concentrated acetic acid. If desired, a reflux may be provided through line 72. The bottom stream out of separator tower 64 is carried through line 74 back to the liquid-liquid contact device 56. A portion of the bottom stream may be carried through a reboiler 76 and returned through line 78 to the separator tower near its bottom. A heat exchanging device may be used to exchange heat between streams 62 and 74 for improved operation.

[0035] In FIG. 2B, the liquid-liquid extractor column is designated 80. It receives a dilute acetic acid feed through line 82. A bottom stream that is relatively pure water leaves the extractor through line 84 and is delivered to the treatment system for waste water or returned to the terephthalic acid plant for reuse there. The top stream consisting primarily of extraction solvent with acetic acid dissolved therein leaves the top of the liquid-liquid extractor 80 through line 86, which delivers it to separator column 88. The overhead line out of separator column 88 is designated 90, and this stream is relatively pure extraction solvent. A condenser 92 is preferably provided in line 90, which delivers the extraction solvent to a point near the bottom of extractor column 80. In separator column 88, the bottom stream is relatively concentrated acetic acid, which leaves through line 92; a reboiler 94 may heat part of this stream for return to the separator column 88 near its bottom. The bulk of the acetic acid in the bottom stream 92 is returned to the terephthalic acid plant for reuse

there.

[0036] FIG. 3 illustrates a preferred embodiment of the invention. In FIG. 3, as well as in FIGS. 4 and 6-10, which show other preferred embodiments of the invention, the items of equipment and lines which are substantially the same as the equipment pieces and lines shown in FIG. 1 are given the same reference number. When lines or equipment are illustrated in these figures which are similar, but differ in some respects either by structure or function, a letter designator such as "A" or "B" may be used in addition to the numerical designator. In the embodiment of FIG. 3, a single distillation column is shown for the primary dehydration step. Those skilled in the art will understand one or more such columns may be used, singularly or stagewise to separate the acids and entrained water from the solvent, and that other kinds of separation equipment may be included.

[0037] In FIG. 3, the acetic acid recovery system includes substantially all of the equipment provided in the prior art unit of FIG. 1 but, in addition, includes an extraction system designated 54. The extraction system may be either like the extraction system 54A of FIG. 2A or the extraction system 54B of FIG. 2B, depending upon the characteristics of the extraction solvent chosen for use in a particular plant. The extraction system 54 is constructed and arranged to receive input to the liquid-liquid extractor through line 50. The extraction unit 54 illustrated in FIG. 3 (and subsequent figures) is one designed for use with a heavy boiling extraction solvent, and is therefore drawn to conform with the extraction system shown in FIG. 2A. For this reason, its extractor unit is designated 56 and the separator is designated 64. The bottom stream out of liquid-liquid extractor 56 is a waste water stream 60 delivered to waste water treatment or, if desired, returned to the terephthalic acid plant for reuse. Furthermore, in accordance with this and succeeding embodiments of the invention, waste water from the extractor 56 may be delivered through line 52A to a high pressure absorber for use therein. The overhead stream out of separator 64 through line 70 is relatively concentrated acetic acid, which is returned to the reactor of the acid plant for reuse. The separator drum 42A in the overhead system from dehydration column 20 may include a coalescing device for separating paraxylene from both the overhead and bottom streams out of the drum, in which case a paraxylene withdrawal line 96 leading to a paraxylene accumulation tank is provided.

[0038] A comparison of the prior art plant of FIG. 1 with the acetic acid recovery system of the invention shown in FIG. 3 reveals that the primary difference is the provision of the extraction system 54 in the unit of the invention. This difference, while it appears to be simple, provides material advantages, because a high acetic acid concentration may be tolerated in the overhead stream from the dehydration column, thus lessening the energy demands of that column, without sacrificing the overall acetic acid recovery level, because the acetic acid in the dehydration column overhead stream is substantially all eventually recovered in the extraction system 54, at a much lower energy cost.

[0039] Attention is now directed to FIG. 4 which shows a further embodiment of the invention. The system of FIG. 4 is also provided with an extraction system 54 for processing overhead condensate delivered from the dehydration column. This embodiment differs from the embodiment of FIG. 3 in that high pressure absorber bottom liquid is delivered through line 26A directly to the extraction system in FIG. 4 instead of being delivered to the dehydration column through line 26 as is the case in FIG. 3. In addition, condensate from a drying system within the terephthalic acid plant is delivered through line 32A to the extraction system 54, instead of being fed to the dehydration column 20 through line 32 as is the case in FIG. 3.

[0040] This modification also represents an improvement, because the high pressure absorber bottom liquid and condensate from the plant drying system both contain a significant concentration of water which, in the embodiment of FIG. 3, is evaporated in the dehydration column. This energy intensive step is circumvented in the embodiment of FIG. 4, since these streams are fed directly to the liquid-liquid extraction column 56 in the extraction system 54.

[0041] FIG. 5 illustrates a flow diagram for a low pressure absorption unit useful in connection with the invention. The low pressure absorption unit of FIG. 5 is designated generally 98, and it includes an absorption tower 100, input for which may be drawn from several sources. These may include an overhead line vent from the dehydration column 44, a vent line from the condenser drum 42 or 42A designated 46, and other vents from the plant designated 102. Some or all of these may be fed through an optional condenser system 104 before being delivered to the absorption column 100 through line 106. Absorption solvent is fed to the absorption column through line 108, and the absorption solvent, now rich in acetic acid, leaves as a bottom stream 110 for delivery to an extraction unit of the kind illustrated in FIGS. 2A and 2B.

[0042] These arrangements are illustrated in the context of an overall system flow sheet in FIG. 6 where the low pressure absorption system is designated 98 and the absorption column is designated 100. As can there be seen, bottom line 110 delivers a stream relatively rich in acetic acid to the liquid-liquid extraction system 54, and in particular to the extraction column thereof 56. The embodiment of the invention illustrated in FIG. 6 is like that shown in FIG. 3, in that high pressure absorber bottom liquid is fed to the dehydration column 20 through line 26 and condensate from a plant drying system is also fed to that dehydration column through line 32.

[0043] FIG. 7 illustrates a flow sheet for an embodiment of the invention which is much like that of FIG. 6, in that it employs both an extraction system 54 and a

low pressure absorption system 98. But it differs from the embodiment of FIG. 6 in that high pressure absorber bottoms liquid is fed directly to the extraction system through line 26A, and condensate from the drying system within the plant is fed to the extraction system 54 through line 32A, thus avoiding the necessity for evaporating the water contained in either of these streams in the dehydration column 20.

[0044] FIGS. 8, 9 and 10 may be considered together. Each shows an embodiment of the invention provided with an extraction system processing condensate from the overhead of the dehydration column designated on each drawing as 54. Each embodiment is also provided with a low pressure absorber system 98, utilizing as inputs streams which, in the prior art system of FIG. 1, are vented to the atmosphere, and which provides an additional stream from its bottom line 110 to the extraction system 54, all in accordance with the invention.

[0045] In FIGS. 8, 9 and 10, the oxidizer of the terephthalic acid plant is fragmentarily shown at 112. The overhead stream from the oxidizer is passed through a condenser 114, and into a separator drum 116. The water draw off liquid from the drum 116 passes as a bottom stream 30 to the dehydration column 20. The overhead from the separation drum passes through line 118 into a high pressure absorption column 120. In the case of FIGS. 8 and 9, the solvent for the high pressure absorber system is delivered through line 52A from the largely water bottom stream from extractor 56. In the case of FIG. 8, the bottom stream from the high pressure absorber is passed through line 122 to the dehydration column 20. In the case of FIG. 9, it passes through bottom line 122A to the extraction system 54. In the case of FIG. 10, the same arrangement is used and the bottom stream passes through line 122 directly to the extraction system 54.

[0046] FIGS. 9 and 10 differ from one another in that in FIG. 10, a phosphine or other selected extraction solvent or absorption solvent is input through line 124 into the high pressure absorber, instead of relying on condensate from the dehydrator overhead through line 52A as appears in FIG. 9.

[0047] From the foregoing, it can be seen that there is considerable flexibility of arrangement of recovery equipment and lines in the acetic acid recovery system of the invention. A guiding principle in taking advantage of such flexibility is the employment of a liquid-liquid extraction system for permitting the presence of a higher concentration in the dehydration column overhead of acetic acid than is tolerable in the prior art, because such extraction system recovers the acetic acid at a low energy cost. The low pressure absorption system provided in accordance with the invention enables the capture of vapour streams that would otherwise be vented to the atmosphere with loss of acetic acid and other organic volatiles, and which creates objectionable atmospheric pollution in many installations. The invention also provides additional flexibility in the handling of the bottom stream from the high pressure absorption system associated with the oxidizer overhead of the terephthalic acid plant, since this may be, but need not be, fed to the dehydration column, but may also be fed to the newly provided extraction system.

Claims

1. A method for separating acetic acid and water, the method being for use in a plant using a water solution of acetic acid, comprising:

   (a) feeding at least one input acetic acid-containing water stream from the plant to a dehydration device (20) and applying heat to the input stream received by the device (20) to separate acetic acid from water in the device (20) to produce thereby an output bottom stream (36) of relatively concentrated acetic acid in water, and an output overhead stream (38) of relatively dilute acetic acid in water,
   (b) condensing acetic acid and water from the said output overhead stream (38) to form an output overhead condensate, and
   (c) feeding the output overhead condensate to a liquid-liquid extraction system (54) and contacting it with a liquid extractant in a contactor (56) to extract acetic acid from the condensate and thereby form a first contactor output stream containing acetic acid and extractant, and a second contactor output stream (60) containing water, feeding the first contactor output stream to an extraction system separator device (64), and separating the acetic acid and the extractant therein to produce an extractant output stream for recycle to the contactor (56) and an acetic acid output stream (70),

   characterised in that the said output overhead stream (38) from the dehydration device (20) has an acetic acid concentration of 2 to 20% by weight.

2. A method as claimed in Claim 1 in which the liquid extractant boils at a temperature higher than acetic acid, and in which the acetic acid output stream from the extraction system separator device (64) is an overhead stream therefrom.

3. A method as claimed in Claim 1 in which the liquid extractant boils at a temperature lower than acetic acid, and in which the acetic acid output stream from the extraction system separator device (88) is a bottoms stream therefrom.

4. A method as claimed in any preceding claim further comprising feeding at least one additional relatively dilute acetic acid stream from the plant to the liquid-

liquid extraction system (54) along with the output overhead stream from the dehydration device (20).

5. A method as claimed in Claim 4 in which the additional relatively dilute acetic acid stream is a bottoms stream (26a) from a high pressure absorber in the plant.

6. A method as claimed in Claim 4 in which the additional relatively dilute acetic acid stream is a condensate stream (32a) from a drying system.

7. A method as claimed in any preceding claim and further comprising contacting an absorption solvent with at least one acetic acid containing vapour stream in a low pressure absorption unit (100) and feeding absorption solvent after contact with the vapour stream to the liquid-liquid extraction system (54).

8. A method as claimed in any preceding claim further comprising contacting an absorbent liquid at high pressure with a plant overhead vapour stream having acetic acid therein, and delivering the absorbent liquid, after contact with the plant overhead vapour stream, to the separator system for processing therein.

9. A method as claimed in Claim 8 in which the absorbent liquid at high pressure is a largely water stream from the liquid-liquid extraction system contactor (56).

10. An acetic acid and water separation system for use in a plant using a water solution of acetic acid, comprising:

(a) a dehydration device (20) equipped to receive at least one input acetic acid-containing water stream from the plant and to apply heat to the input stream received by the device (20) to separate water from acetic acid in the device (20) thereby to produce an output bottom stream (36) of relatively concentrated acetic acid in water, and an output overhead stream (38) of acetic acid in water having an acetic acid concentration of 2 to 20% by weight,
(b) condensing means (40) to liquify acetic acid and water from the output overhead stream (38) to form an output overhead condensate, and
(c) a liquid-liquid extraction system (54) comprising:

(i) a contactor (56) to receive the output overhead condensate and contact it with a liquid extractant to extract acetic acid from the condensate and thereby form a first contactor output stream containing acetic acid and extractant, and a second contactor output stream (60) containing water, and
(ii) an extraction system separator device (64) equipped to receive the said first contactor output stream and separate the acetic acid and the extractant therein to produce an extractant output stream for recycle to the contactor (56) and an acetic acid output stream (70),

characterised in that means are provided for feeding at least one additional relatively dilute acetic acid stream from the plant to the liquid-liquid extraction system (54) along with the output overhead condensate from the dehydration device (20).

11. A system as claimed in Claim 10 in which the liquid extractant boils at a temperature higher than acetic acid, and in which the acetic acid output stream from the extraction system separator device (64) is an overhead stream therefrom.

12. A system as claimed in Claim 10 in which the liquid extractant boils at a temperature lower than acetic acid, and in which the acetic acid output stream from said extraction system separator device (88) is a bottoms stream therefrom.

13. A system as claimed in any of claims 10 to 12 in which the additional relatively dilute acetic acid stream is a bottoms stream (26a) from a high pressure absorber in the plant.

14. A system as claimed in any of claims 10 to 12 in which the additional relatively dilute acetic acid stream is a condensate stream (32a) from a drying system.

15. A system as claimed in any of claims 10 to 14 and further comprising a low pressure absorption unit (100) adapted to contact an absorption solvent with at least one acetic acid containing vapour stream from the acetic acid and water separation system, and further adapted to feed absorption solvent after contact with the vapour stream to the liquid-liquid extraction system (54).

16. A system as claimed in any of claims 10 to 15 and further comprising a high pressure absorber system (116) equipped to contact an absorbent liquid at high pressure with a plant overhead vapour stream having acetic acid therein, and means for delivering the absorbent liquid, after contact with the plant overhead vapour stream, to the separator system for processing therein.

17. A system as claimed in Claim 16 in which the absorbent liquid at high pressure is a largely water stream from the liquid-liquid extraction system contactor (56).

18. A system as claimed in any of claims 10 to 17 in combination with and as part of a plant using a water solution of acetic acid.

19. A system as claimed in Claim 18 in which the said plant is a plant for the production of terephthalic acid.

Patentansprüche

1. Verfahren zum Trennen von Essigsäure und Wasser, das zur Verwendung in einer Anlage bestimmt ist, in der eine wäßrige Essigsäurelösung verwendet wird, umfassend:

   a) Zuführen von wenigstens einem essigsäurehaltigen Eingangswasserstrom von der Anlage zu einer Dehydratisierungsvorrichtung (20) und Beaufschlagen des von der Vorrichtung (20) erhaltenen Eingangsstroms mit Wärme, um die Essigsäure von Wasser in der Vorrichtung (20) abzuscheiden, um dadurch einen Bodenausgangsstrom (36) mit relativ hoher Essigsäurekonzentration in Wasser und einen Überkopfausgangsstrom (38) mit relativ niedriger Essigsäurekonzentration in Wasser zu erzeugen,
   b) Kondensieren von Essigsäure und Wasser von dem genannten Überkopfausgangsstrom (38) zur Bildung eines Überkopfausgangskondensats, und
   c) Zuführen des Überkopfausgangskondensats zu einem Flüssig-flüssig-Extraktionssystem (54) und In-Kontakt-Bringen desselben mit einem Flüssigkeitsextraktionsmittel in einem Kontaktor (56), um Essigsäure aus dem Kondensat zu extrahieren und so einen ersten Kontaktorausgangsstrom, der Essigsäure und Extraktionsmittel enthält, und einen zweiten Kontaktorausgangsstrom (60) zu bilden, der Wasser enthält, Zuführen des ersten Kontaktorausgangsstroms zu einer Extraktionssystem-Abscheidungsvorrichtung (64), und Trennen der Essigsäure und des Extraktionsmittels darin, um einen Extraktionsmittel-Ausgangsstrom zur Rückführung zu dem Kontaktor (56) und einen Essigsäure-Ausgangsstrom (70) zu erzeugen,

   dadurch gekennzeichnet, daß der genannte Überkopfausgangsstrom (38) von der Dehydratisierungsvorrichtung (20) eine Essigsäurekonzentration von 2 bis 20 Gew.-% hat.

2. Verfahren nach Anspruch 1, bei dem der Siedepunkt des Flüssigkeitsextraktionsmittels höher ist als der der Essigsäure, und bei dem der Essigsäure-Ausgangsstrom von der Extraktionssystem-Abscheidervorrichtung (64) ein Überkopfstrom davon ist.

3. Verfahren nach Anspruch 1, bei dem der Siedepunkt des Flüssigkeitsextraktionsmittels niedriger ist als der der Essigsäure, und bei dem der Essigsäure-Ausgangsstrom von der Extraktionssystem-Abscheidervorrichtung (88) ein Bodenstrom davon ist.

4. Verfahren nach einem der vorherigen Ansprüche, ferner umfassend das Zuführen von wenigstens einem zusätzlichen relativ schwach konzentrierten Essigsäurestrom von der Anlage zu dem Flüssig-flüssig-Extraktionssystem (54) zusammen mit dem Überkopfausgangsstrom von der Dehydratisierungsvorrichtung (20).

5. Verfahren nach Anspruch 4, bei dem der zusätzliche relativ schwach konzentrierte Essigsäurestrom ein Bodenstrom (26a) von einem Hochdruckabsorber in der Anlage ist.

6. Verfahren nach Anspruch 4, bei dem der zusätzliche relativ schwach konzentrierte Essigsäurestrom ein Kondensatstrom (32a) von einem Trocknungssystem ist.

7. Verfahren nach einem der vorherigen Ansprüche, ferner umfassend das In-Kontakt-Bringen eines Absorptionslösemittels mit wenigstens einem essigsäurehaltigen Dampfstrom in einer Niederdruck-Absorptionseinheit (100) und Zuführen von Absorptionslösemittel nach dem Kontakt mit dem Dampfstrom zu dem Flüssig-flüssig-Extraktionssystem (54).

8. Verfahren nach einem der vorherigen Ansprüche, ferner umfassend das In-Kontakt-Bringen einer Absorptionsflüssigkeit mit hohem Druck mit einem Anlagen-Überkopfdampfstrom mit darin enthaltener Essigsäure und Zuführen der Absorptionsmittelflüssigkeit nach dem Kontakt mit dem Anlagen-Überkopfdampfstrom zu dem Abscheidersystem zur Verarbeitung darin.

9. Verfahren nach Anspruch 8, bei dem die Hochdruck-Absorptionsmittelflüssigkeit ein weitgehend aus Wasser bestehender Strom von dem Flüssig-flüssigExtraktionssystem-Kontaktor (56) ist.

10. Essigsäure- und Wassertrennsystem für die Verwendung in einer Anlage mit einer wäßrigen Essigsäurelösung, umfassend:

a) eine Dehydratisierungsvorrichtung (20), die so ausgestattet ist, daß sie wenigstens einen essigsäurehaltigen Eingangswasserstrom von der Anlage empfängt und den Eingangsstrom von der Vorrichtung (20) mit Wärme beaufschlagt, um Wasser aus der Essigsäure in der Vorrichtung (20) abzuscheiden, um so einen Bodenausgangsstrom (36) mit relativ hoher Essigsäurekonzentration in Wasser und einen Überkopfausgangsstrom (38) von Essigsäure in Wasser mit einer Essigsäurekonzentration von 2 bis 20 Gew.-% zu erzeugen,

b) ein Kondensationsmittel (40) zum Verflüssigen von Essigsäure und Wasser von dem Überkopfausgangsstrom (38) zur Bildung eines Überkopfausgangskondensats, und

c) ein Flüssig-flüssig-Extraktionssystem (54), umfassend:

(i) einen Kontaktor (56) zum Empfangen des Überkopfausgangskondensats und In-Kontakt-Bringen desselben mit einem Flüssigkeitsextraktionsmittel zum Extrahieren von Essigsäure aus dem Kondensat und um so einen ersten Kontaktor-Ausgangsstrom, der Essigsäure und Extraktionsmittel enthält, und einen zweiten Kontaktorausgangsstrom (60) zu bilden, der Wasser enthält, und

(ii) eine Extraktionssystem-Abscheidervorrichtung (64), die so ausgestattet ist, daß sie den genannten ersten Kontaktorausgangsstrom empfängt und die Essigsäure und das Extraktionsmittel darin abscheidet, um einen Extraktionsmittel-Ausgangsstrom zur Rückführung zu dem Kontaktor (56) und einen Essigsäure-Ausgangsstrom (70) zu erzeugen,

dadurch gekennzeichnet, daß Mittel zum Zuführen von wenigstens einem zusätzlichen relativ schwach konzentrierten Essigsäurestrom von der Anlage zu dem Flüssig-flüssig-Extraktionssystem (54) zusammen mit dem Überkopfausgangskondensat von der Dehydratisierungsvorrichtung (20) vorgesehen sind.

11. System nach Anspruch 10, bei dem der Siedepunkt des Flüssigkeitsextraktionsmittels höher ist als der der Essigsäure, und bei dem der Essigsäure-Ausgangsstrom von der Extraktionssystem-Abscheidervorrichtung (64) ein Überkopfstrom davon ist.

12. System nach Anspruch 10, bei dem der Siedepunkt des Flüssigkeitsextraktionsmittels niedriger ist als der der Essigsäure, und bei dem der Essigsäure-Ausgangsstrom von der genannten Extraktionssystem-Abscheidervorrichtung (88) ein Bodenstrom davon ist.

13. System nach einem der Ansprüche 10 bis 12, bei dem der zusätzliche relativ schwach konzentrierte Essigsäurestrom ein Bodenstrom (26a) von einem Hochdruckabsorber in der Anlage ist.

14. System nach einem der Ansprüche 10 bis 12, bei dem der zusätzliche relativ schwach konzentrierte Essigsäurestrom ein Kondensatstrom (32a) von einem Trocknungssystem ist.

15. System nach einem der Ansprüche 10 bis 14, ferner umfassend eine Niederdruck-Absorptionseinheit (100) zum In-Kontakt-Bringen eines Absorptionslösemittels mit wenigstens einem essigsäurehaltigen Dampfstrom von dem Essigsäure- und Wasserabscheidungssystem und ferner zum Zuführen von Absorptionslösemittel nach dem Kontakt mit dem Dampfsystem zu dem Flüssig-flüssig-Extraktionssystem (54).

16. System nach einem der Ansprüche 10 bis 15, ferner umfassend ein Hochdruck-Absorbersystem (116) zum In-Kontakt-Bringen einer Absorptionsmittelflüssigkeit mit hohem Druck mit einem Anlagen-Überkopfdampfstrom mit darin enthaltener Essigsäure, und Mittel zum Zuführen der Absorptionsflüssigkeit nach dem Kontakt mit dem Anlagen-Überkopfstrom zu dem Abscheidersystem zur Verarbeitung darin.

17. System nach Anspruch 16, bei dem die Hochdruck-Absorptionsflüssigkeit ein weitgehend aus Wasser bestehender Strom von dem Flüssig-flüssig-Extraktionssystem-Kontaktor (56) ist.

18. System nach einem der Ansprüche 10 bis 17 in Kombination mit und als Bestandteil einer Anlage, die eine wäßrige Essigsäurelösung verwendet.

19. System nach Anspruch 18, bei dem die genannte Anlage eine Anlage für die Herstellung von Terephthalsäure ist.

Revendications

1. Une méthode de séparation de l'acide acétique et de l'eau, la méthode étant conçue pour utilisation dans une usine utilisant une solution d'acide acétique dans l'eau, comportant :

(a) introduction d'au moins une veine d'entrée d'eau contenant de l'acide acétique à partir de l'usine vers un dispositif de déshydratation (20) et application de chaleur à la veine d'entrée reçue par le dispositif (20) pour séparer l'acide acétique de l'eau dans le dispositif (20) de

manière à produire une veine inférieure de sortie (36) d'acide acétique relativement concentré dans l'eau, et une veine de sortie supérieure (38) d'acide acétique relativement dilué dans l'eau ;

(b) condensation de l'acide acétique et de l'eau à partir de ladite veine de sortie supérieure (38) pour former un condensat supérieur de sortie, et

(c) envoi du condensat supérieur de sortie vers un système d'extraction liquide-liquide (54) et mise en contact de ce condensat avec un produit d'épuisement liquide dans un contacteur (56) pour extraire l'acide acétique du condensat afin de former une première veine de sortie de contacteur contenant de l'acide acétique et un produit d'épuisement, et une seconde veine de sortie de contacteur (60) contenant de l'eau, envoi de la première veine de sortie du contacteur vers un dispositif séparateur du système d'extraction (64), et séparation de l'acide acétique et du produit d'épuisement qu'il contient pour produire une veine de sortie de produit d'épuisement pour recyclage vers le contacteur (56) et une veine de sortie d'acide acétique (70) ;

caractérisée en ce que ladite veine supérieure de sortie (38) du dispositif de déshydratation (20) a une concentration d'acide acétique de 2 à 20% en poids.

2. Une méthode selon la Revendication 1, selon laquelle le produit d'épuisement liquide bout à une température supérieure à celle de l'acide acétique, et selon laquelle la veine de sortie d'acide acétique du dispositif séparateur du système d'extraction (64) est une veine supérieure de ce dispositif.

3. Une méthode selon la Revendication 1, selon laquelle le produit d'épuisement liquide bout à une température inférieure à celle de l'acide acétique, et selon laquelle la veine de sortie d'acide acétique du dispositif séparateur du système d'extraction (88) est une veine inférieure de celui-ci.

4. Une méthode selon l'une quelconque des revendications précédentes, qui comporte de plus l'envoi d'au moins une veine additionnelle d'acide acétique relativement dilué à partir de l'usine vers le système d'extraction liquide-liquide (54) en même temps que la veine supérieure de sortie du dispositif de déshydratation (20).

5. Une méthode selon la Revendication 4, selon laquelle la veine additionnelle d'acide acétique relativement dilué est une veine inférieure (26a) qui provient d'un absorbeur à haute pression de l'usine.

6. Une méthode selon la Revendication 4, selon laquelle la veine additionnelle d'acide acétique relativement dilué est une veine de condensat (32a) qui provient d'un système sécheur.

7. Une méthode selon l'une quelconque des revendications précédentes qui comporte de plus la mise en contact d'un solvant d'absorption avec au moins une veine de vapeur contenant de l'acide acétique dans une unité d'absorption à basse pression (100) et l'envoi du solvant d'absorption après le contact avec la veine de vapeur vers le système d'extraction liquide-liquide (54).

8. Une méthode selon l'une quelconque des revendications précédentes, qui comprend de plus la mise en contact d'un liquide absorbant à haute pression avec une veine de vapeur supérieure de l'usine contenant de l'acide acétique, et l'envoi du liquide absorbant, après le contact avec la veine de vapeur supérieure de l'usine, vers le système séparateur pour son traitement à l'intérieur.

9. Une méthode selon la Revendication 8, selon laquelle le liquide absorbant à haute pression est une veine largement composée d'eau qui provient du contacteur du système d'extraction liquide-liquide (56).

10. Un système de séparation d'acide acétique et d'eau pour utilisation dans une usine qui utilise une solution d'acide acétique dans l'eau, comportant :

(a) un dispositif de déshydratation (20) équipé pour recevoir au moins une veine d'entrée d'eau contenant de l'acide acétique provenant de l'usine et pour appliquer de la chaleur à la veine d'entrée reçue par le dispositif (20) de manière à séparer l'eau de l'acide acétique dans le dispositif (20) afin de produire une veine inférieure de sortie (36) d'acide acétique relativement concentré dans l'eau et une veine supérieure de sortie (38) d'acide acétique dans l'eau ayant une concentration d'acide acétique de 2 à 20% en poids ;

(b) des moyens de condensation (40) pour liquéfier l'acide acétique et l'eau de la veine supérieure de sortie (38) pour former un condensat supérieur de sortie, et

(c) un système d'extraction liquide-liquide (54) comportant :

(i) un contacteur (56) pour recevoir le condensat supérieur de sortie et le mettre en contact avec un produit d'épuisement liquide pour extraire l'acide acétique du condensat et pour former ainsi une première veine de sortie du contacteur conte-

nant de l'acide acétique et du produit d'épuisement et une seconde veine de sortie du contacteur (60) contenant de l'eau, et

(ii) un dispositif séparateur du système d'extraction (64) équipé pour recevoir ladite première veine de sortie du contacteur et séparer l'acide acétique et le produit d'épuisement qu'elle contient pour produire une veine de sortie de produit d'épuisement pour recyclage vers le contacteur (56) et une veine de sortie d'acide acétique (70) ;

caractérisé en ce que les moyens sont prévus pour envoyer au moins une veine additionnelle d'acide acétique relativement dilué à partir de l'usine vers le système d'extraction liquide-liquide (54) en même temps que le condensat supérieur de sortie qui provient du dispositif de déshydratation (20).

11. Un système selon la Revendication 10, dans lequel le produit d'épuisement liquide bout à une température supérieure à celle de l'acide acétique et dans lequel la veine de sortie d'acide acétique du dispositif séparateur du système d'extraction (64) est une veine supérieure de celui-ci.

12. Un système selon la Revendication 10, dans lequel le produit d'épuisement liquide bout à une température inférieure à celle de l'acide acétique, et dans lequel la veine de sortie d'acide acétique du dit dispositif séparateur du système d'extraction (88) est une veine inférieure de celui-ci.

13. Un système selon l'une quelconque des Revendications 10 à 12, dans lequel la veine additionnelle d'acide acétique relativement dilué est une veine inférieure (26a) qui provient d'un absorbeur à haute pression dans l'usine.

14. Un système selon l'une quelconque des Revendications 10 à 12, dans lequel la veine additionnelle d'acide acétique relativement dilué est une veine de condensat (32a) qui provient d'un système sécheur.

15. Un système selon l'une quelconque des revendications 10 à 14 et qui comprend de plus une unité d'absorption à basse pression (100) adaptée pour la mise en contact d'un solvant d'absorption avec au moins une veine de vapeur contenant de l'acide acétique qui provient du système de séparation d'acide acétique et d'eau, et adaptée de plus pour envoyer le solvant d'absorption, après son contact avec la veine de vapeur, dans le système d'extraction liquide-liquide (54).

16. Un système selon l'une quelconque des Revendications 10 à 15 et qui comprend de plus un système absorbeur à haute pression (116) équipé pour mettre en contact un liquide absorbant à haute pression avec une veine de vapeur supérieure de l'usine contenant de l'acide acétique et des moyens d'envoi du liquide absorbant, après le contact avec la veine de vapeur supérieure de l'usine, vers le système séparateur pour le traitement à l'intérieur de celui-ci.

17. Un système selon la Revendication 16, dans lequel le liquide absorbant à haute pression est une veine contenant principalement de l'eau qui provient du contacteur du système d'extraction liquide-liquide (56).

18. Un système selon l'une quelconque des Revendications 10 à 17 conjointement avec une usine utilisant une solution d'acide acétique dans l'eau et qui fait partie de cette usine.

19. Un système selon la Revendication 18, dans lequel ladite usine est une usine de production d'acide téréphtalique.

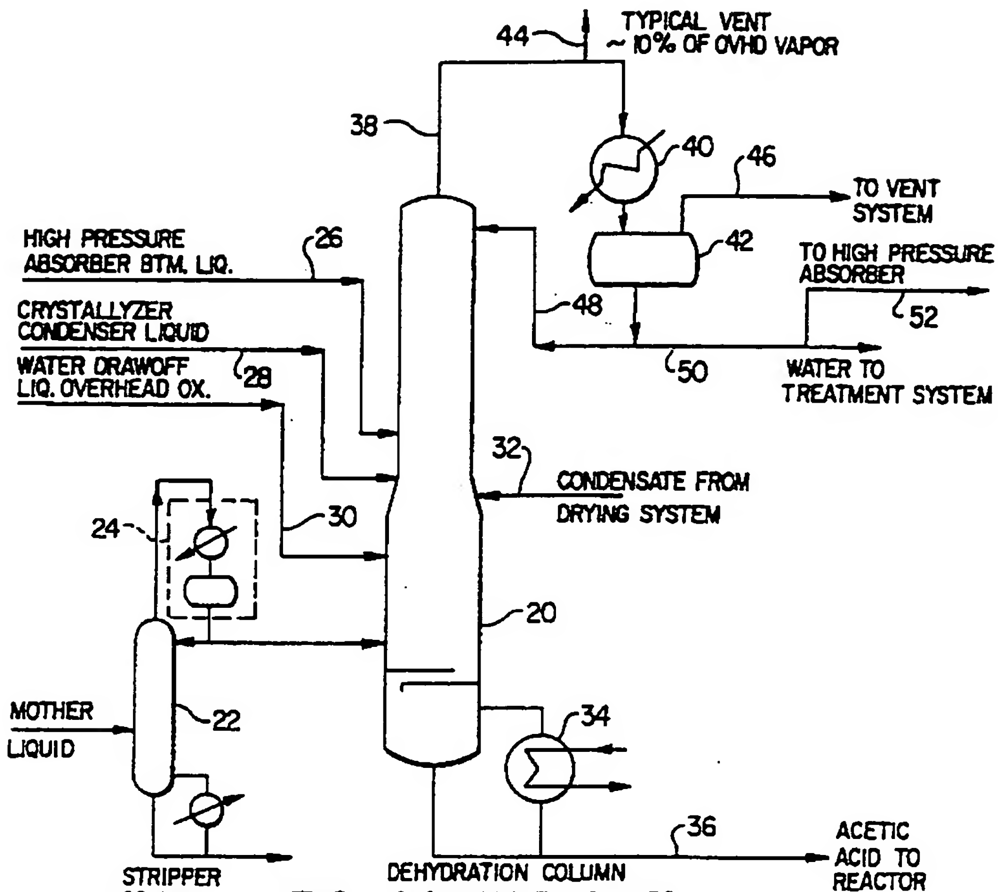

FIG. 1 (PRIOR ART)

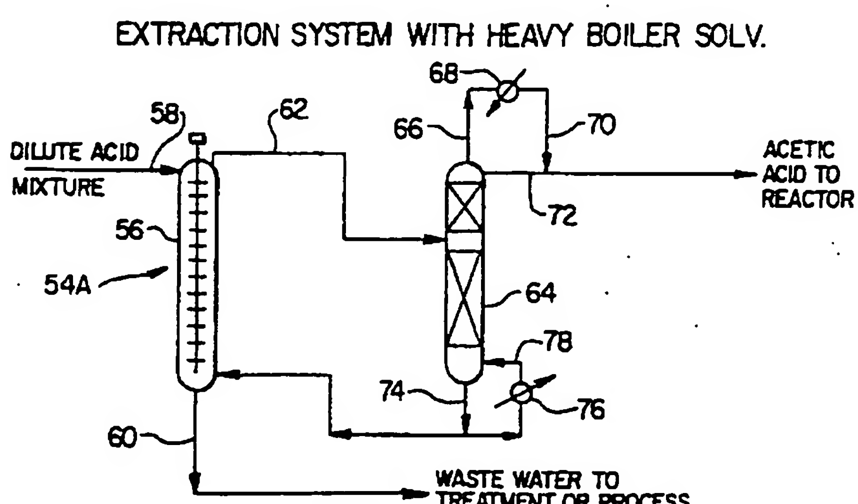

FIG. 2A

EXTRACTION SYSTEM WITH LIGHT BOILER SOLV.

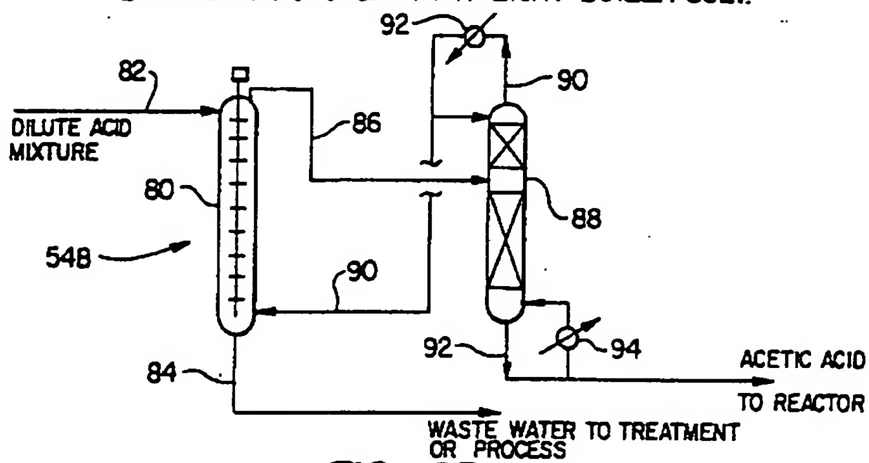

FIG. 2B

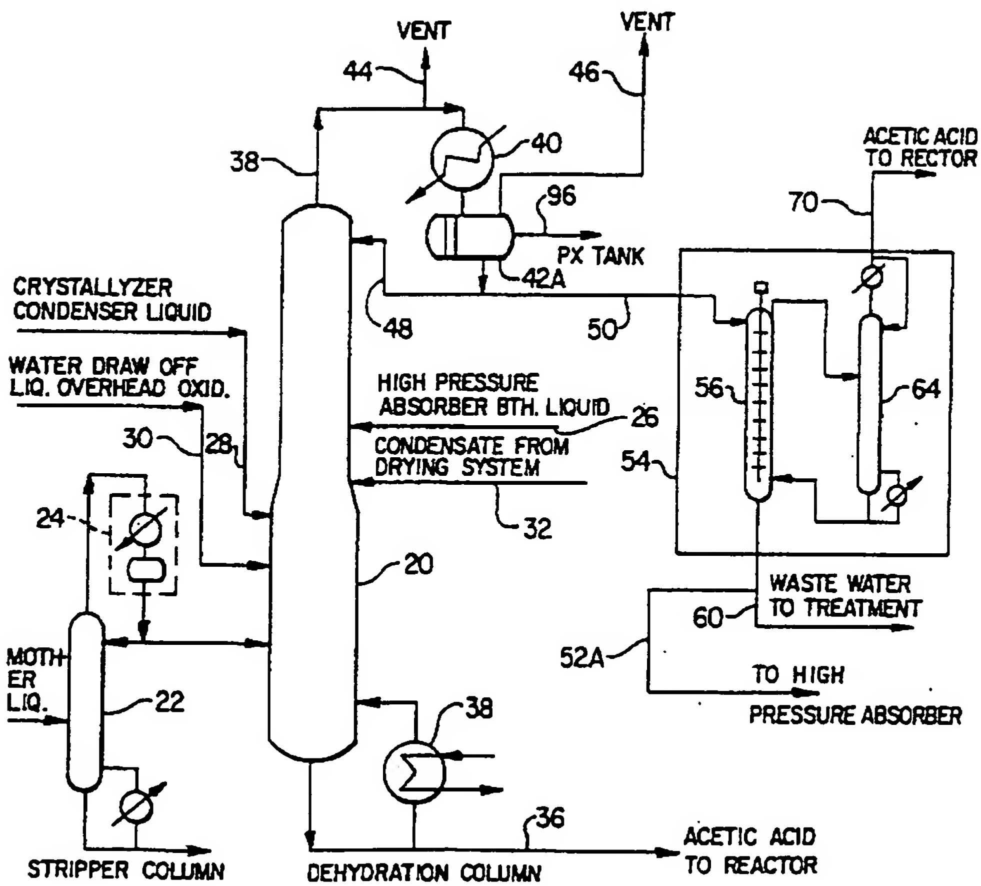

FIG. 3 (EXTRACTION SYSTEM)

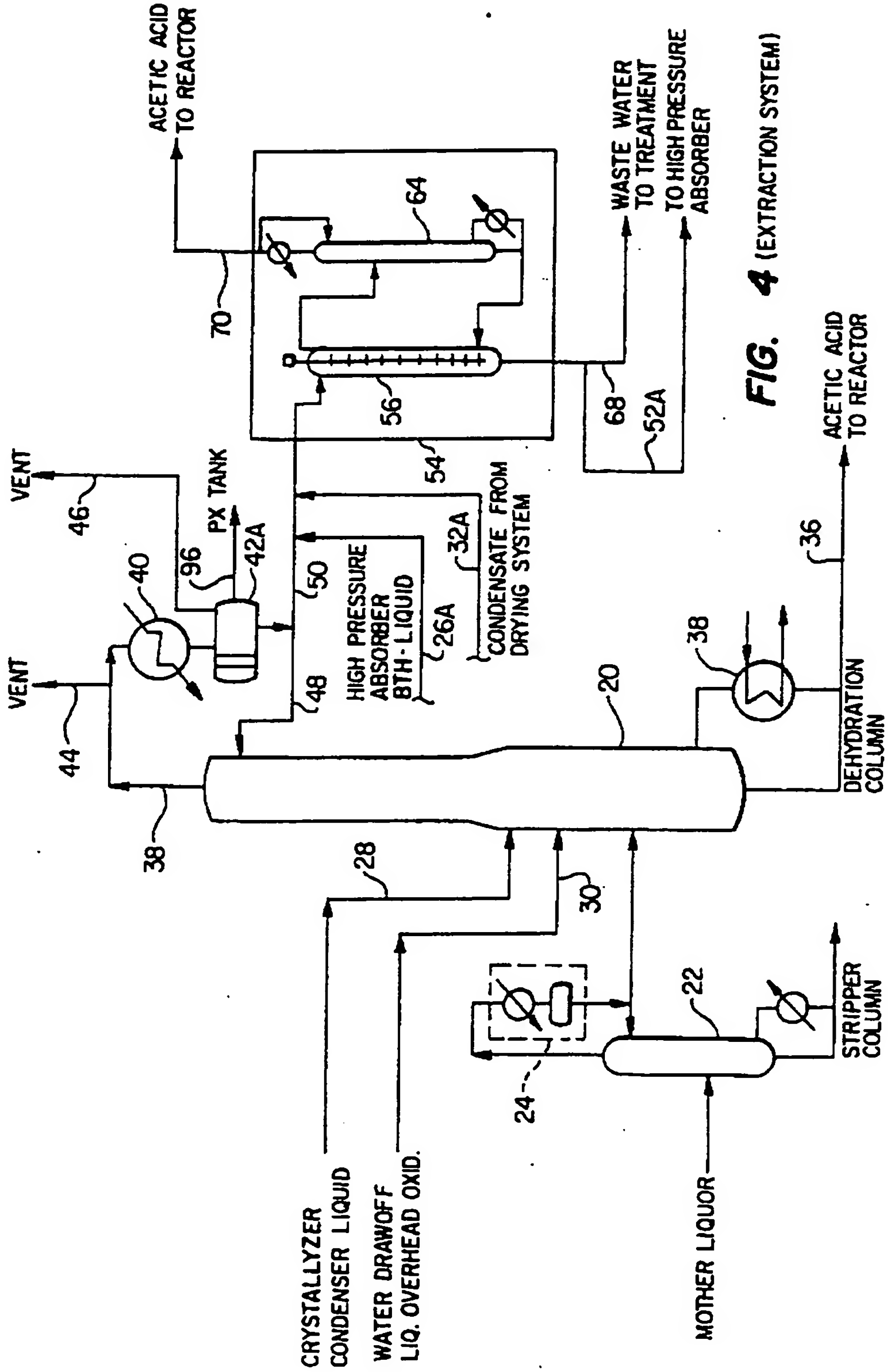

FIG. 4 (EXTRACTION SYSTEM)

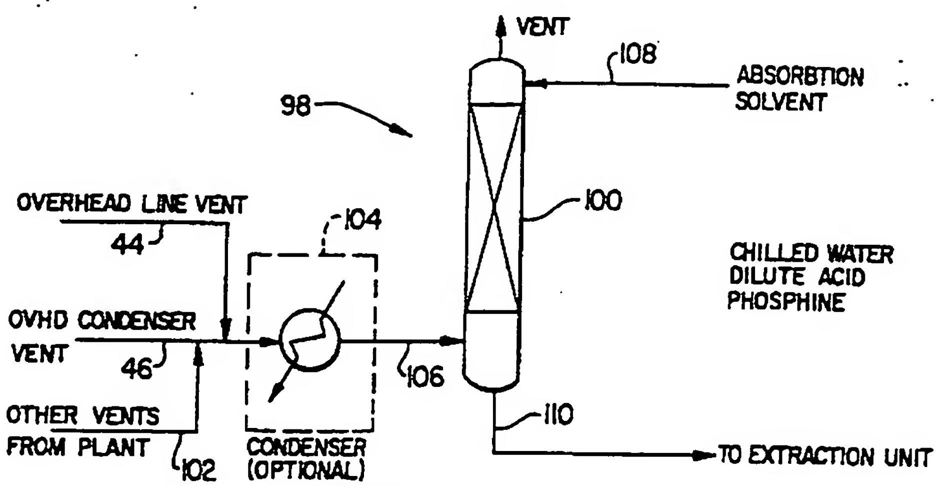

FIG. 5

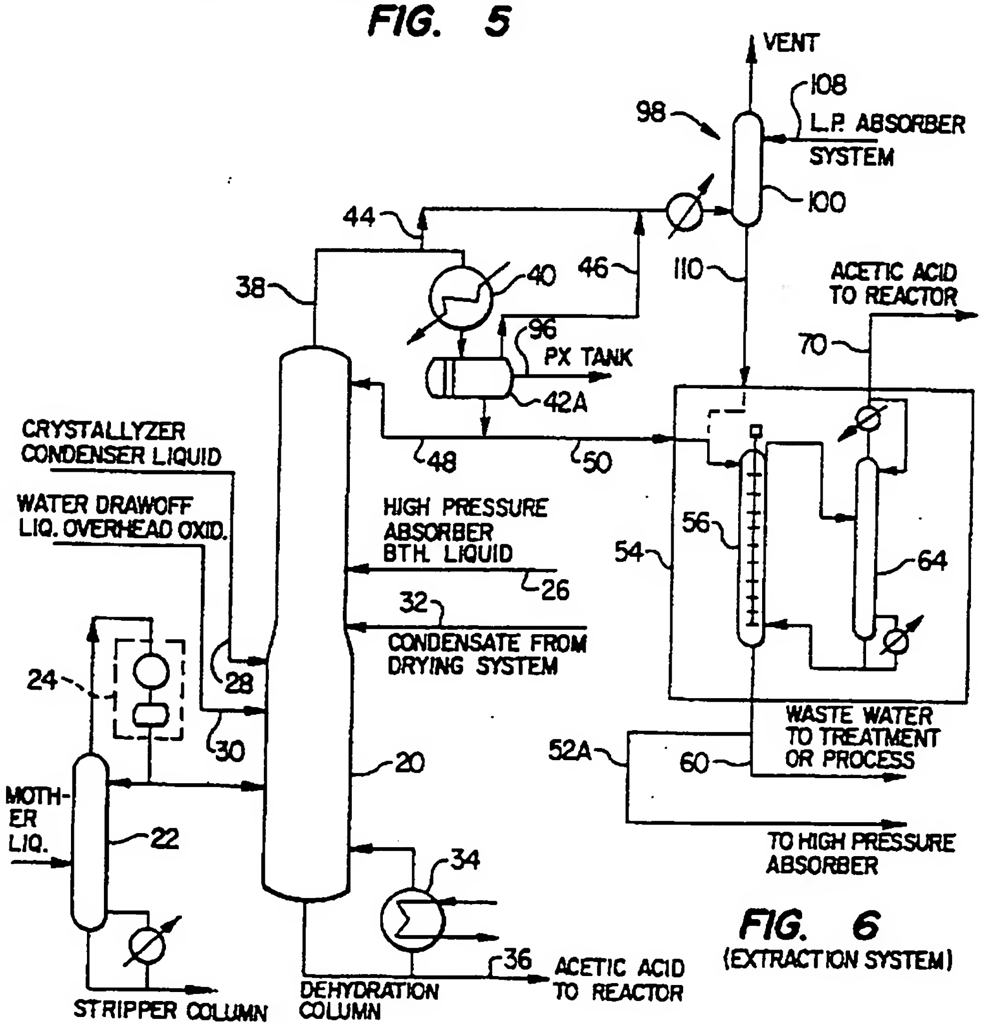

FIG. 6 (EXTRACTION SYSTEM)

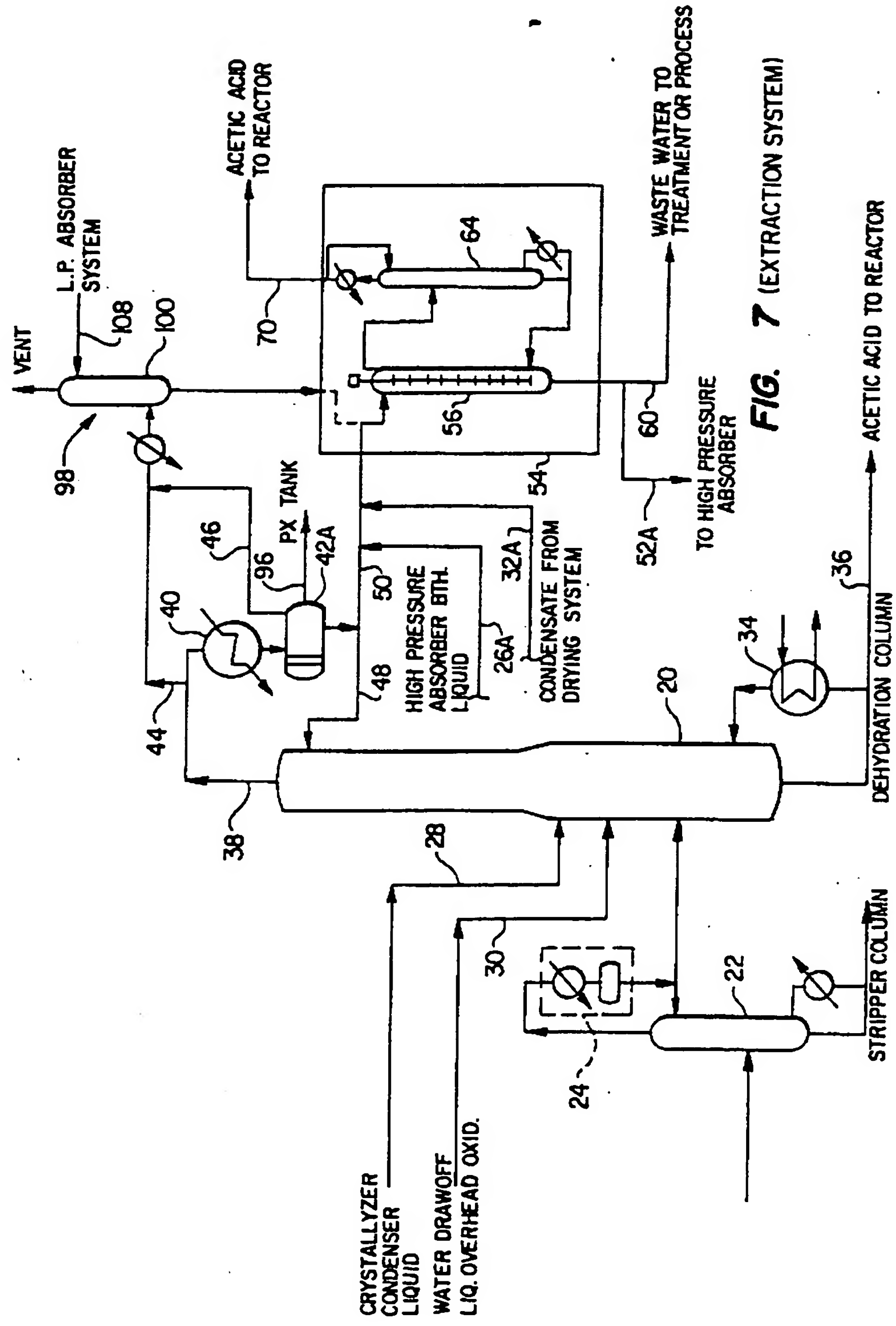

FIG. 7 (EXTRACTION SYSTEM)

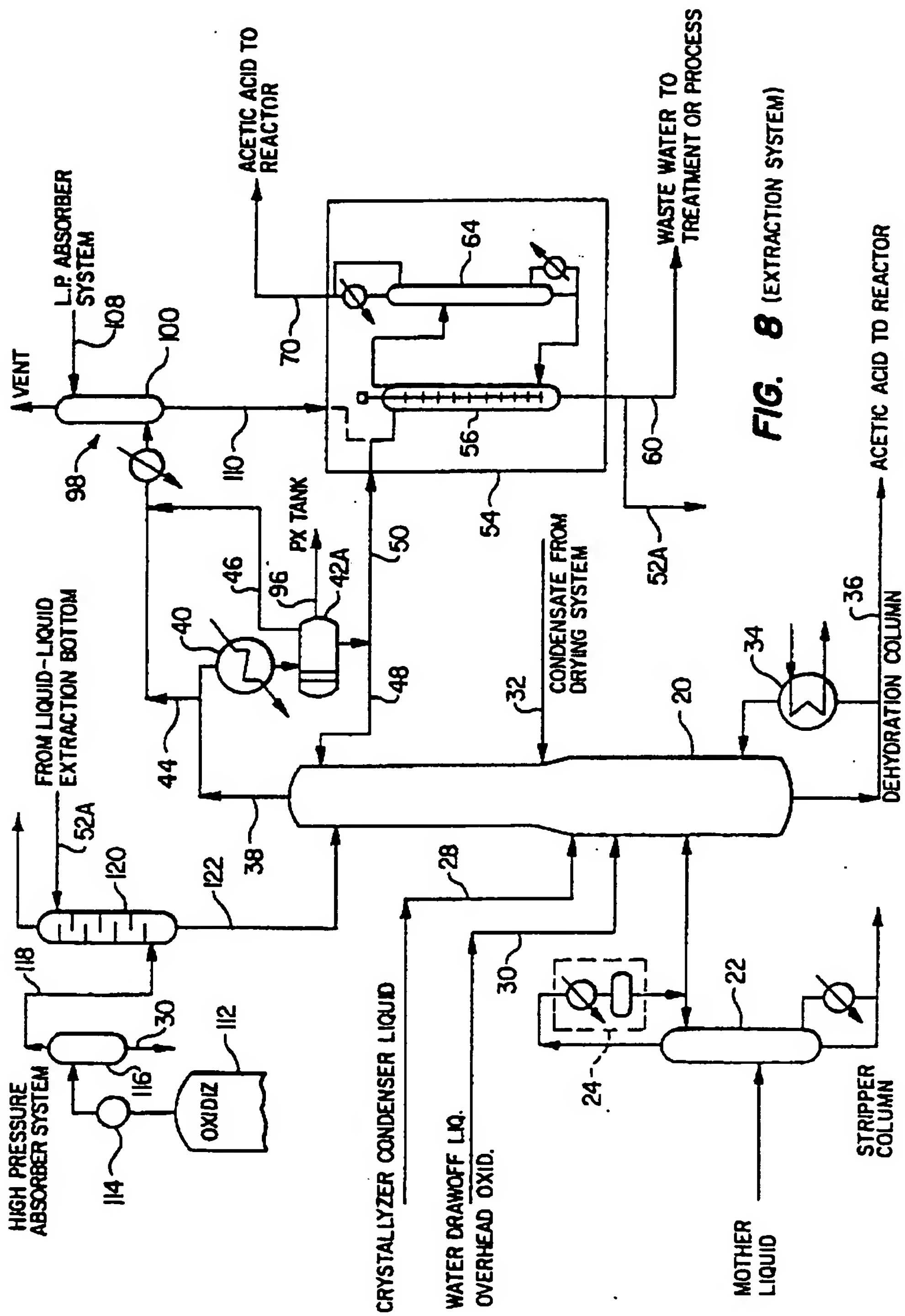

FIG. 8 (EXTRACTION SYSTEM)

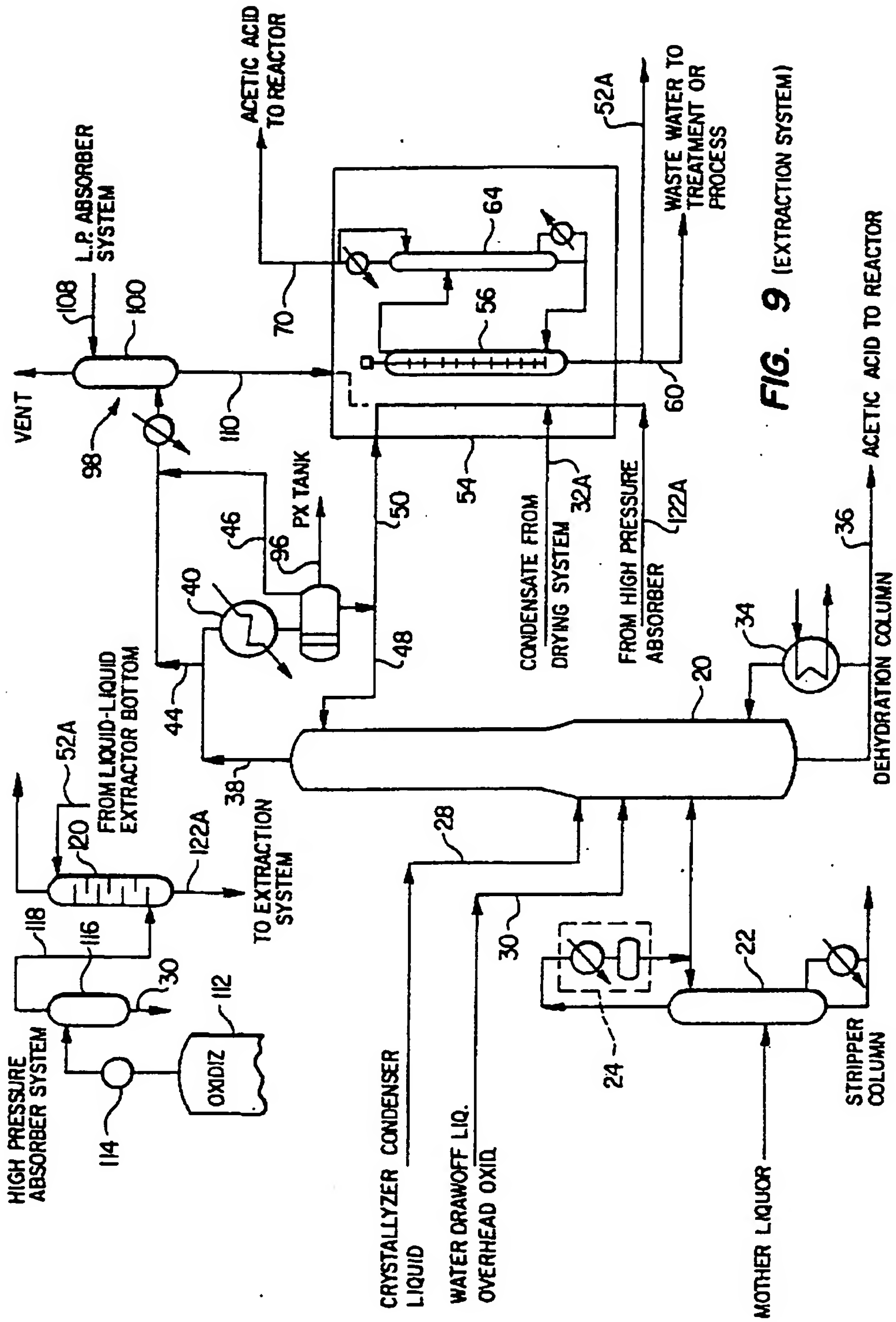

FIG. 9 (EXTRACTION SYSTEM)

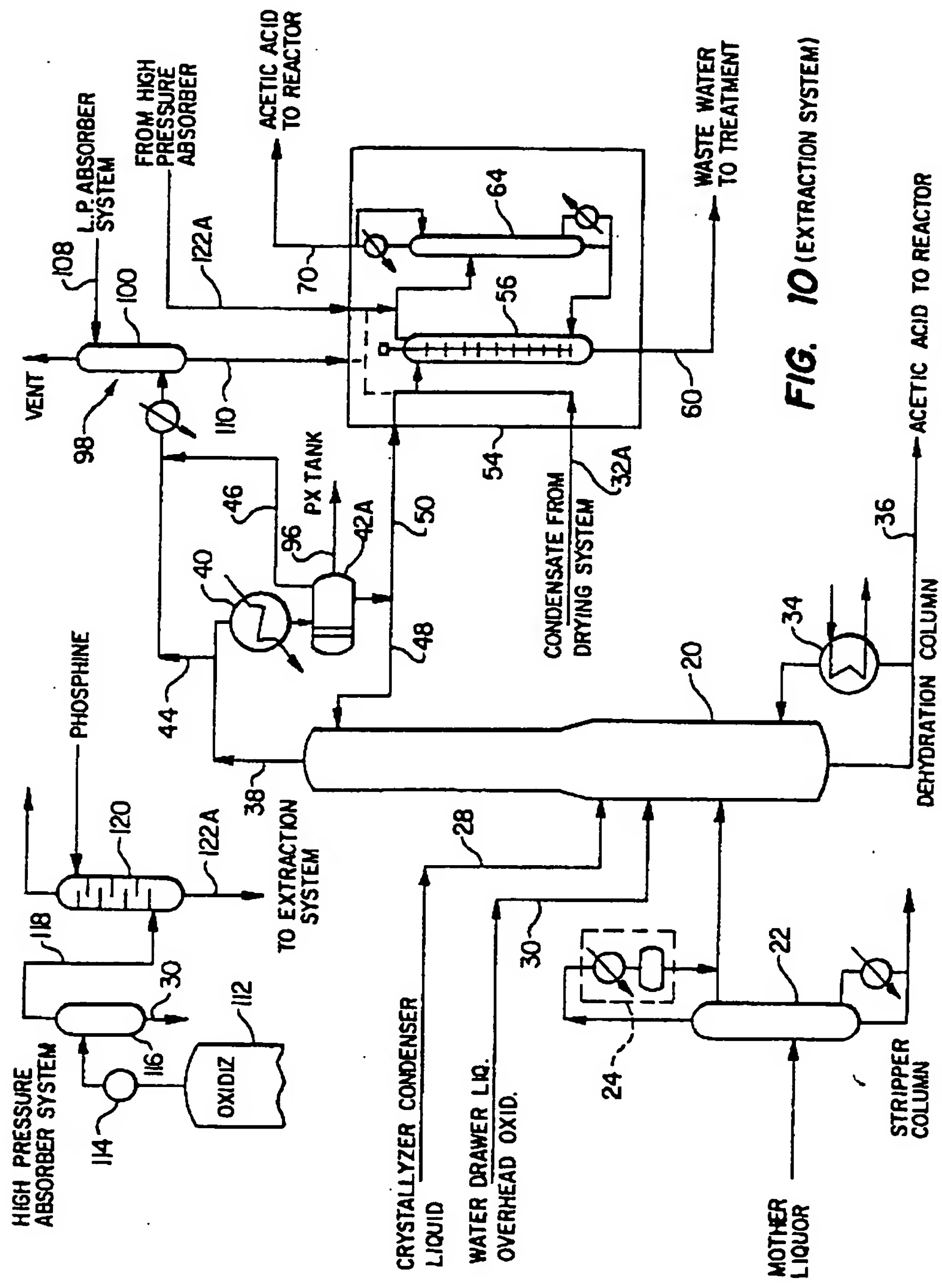

FIG. 10 (EXTRACTION SYSTEM)